

## Comparison of the Microstructure & Segregation behavior between SA508 Gr.3 & SA508 Gr.4N High Strength Low Alloy RPV Steel

Sang Gyu Park<sup>a</sup>, Min-Chul Kim<sup>b</sup>, Bong Sang Lee<sup>b</sup>, Dang Moon Wee<sup>a</sup>

<sup>a</sup> KAIST, Guseong-dong, Yuseong-gu, Daejeon, 305-701, Republic of Korea\*

<sup>b</sup> KAERI, Nuclear Materials Research Division., Deogjin-dong, Yuseong-gu, Daejeon, 305-353, Republic of Korea  
sg121243 @ kaist.ac.kr

### 1. Introduction

It is generally known that SA508 Gr.4N low alloy steel has an improved fracture toughness and strength, compared to commercial low alloy steels such as SA508 Gr.3 which have lower than 1% Ni. Higher strength and fracture toughness of low alloy steels could be achieved by adding Ni and Cr. So there are several researches on SA508 Gr.4N low alloy steel for a RPV application[1,2]. The operation temperature and time of a reactor pressure vessel is more than 300 and over 40 years. Therefore, in order to apply the SA508 Gr.4N low alloy steel for a reactor pressure vessel, it requires a phase stability in the high temperature range including temper embrittlement resistance. S. Raoul [3] reported that the susceptibility to temper embrittlement was increasing a function of the cooling rate in SA533 steel, which suggests the martensitic microstructures resulting from increased cooling rates are more susceptible to temper embrittlement. However, this result has not been proved yet. So comparison was made between the temper embrittlement behaviors of SA508 Gr.3 and Gr.4N low alloy steel with a viewpoint of boundary features, which have different microstructures of tempered bainite(SA508 Gr.3) and tempered martensite(SA508 Gr.4N).

In this study, we have compared temper embrittlement behaviors of SA508 Gr.3 & SA508 Gr.4N low alloy steel. The mechanical properties of these low alloy steels after a long-term heat treatment(450 , 2000hr) were evaluated. Then, the images of the fracture surfaces were observed and grain boundary segregation was analyzed by AES. In order to compare the misorientation distributions of two model alloys, the grain boundary structures of the low alloy steels with EBSD were measured.

### 2. Experimental Procedure

Two model alloys of SA508 Gr.3 and SA508 Gr.4N low alloy steels were selected for this study. The chemical compositions of the steels are given in Table 1. The model alloy of KL3 has a similar composition to commercial RPV steel used in KSNP, and KL4 with a typical composition of the SA508 Gr.4N steel was arranged as a reference alloy within ASME specified composition. Both alloys were austenitized at 880 for 2 hours followed by an air cooling, and then tempered at 660 for 10 hours. After the tempering process, the model alloys were treated at 450 for 2000 hours,

which can reveal the temper embrittlement phenomena efficiently[4].

Impact transition curves were obtained using standard Charpy V-notched specimens and using an SATEC-S1 impact test machine with maximum capacity of 406J in a temperature range of -196 to 150 . The index temperatures were determined from fitted Charpy curves as the temperature corresponding to the Charpy energy values of 48J and 68J.

The observations of the fractures were conducted using a scanning electron microscope (SEM). The specimens were examined using an SEM-6300 scanning electron microscope. Auger electron spectroscopy was used to monitor the grain boundary segregation in the model alloy. All samples were fractured at low temperature (lower than -150 ) in  $2 \times 10^{-10}$  torr, and the fracture surfaces were analyzed at 5kV. A ULVAC PHI 700 auger electron microscope was employed for the analysis.

The grain boundary segregation behavior was evaluated by Secondary Ion Mass Spectroscopy (SIMS). The specimens were prepared in a disk 1mm in diameter and 2mm in thickness. They were analyzed by IMS-6f secondary ion mass spectroscopy. The grain boundary structures were observed by Electron Back-Scattered Diffraction (EBSD) using a JSM-700F field-emission scanning electron microscope.

Table 1. Chemical compositions of steels. (wt%)

	C	Mn	Ni	Cr	P	Fe
KL3	0.20	1.40	0.90	0.15	.002	Bal.
KL4	0.20	0.30	3.64	1.80	.002	Bal.

### 3. Experimental Results and Discussion

Fig. 1 shows the Charpy impact test results. From the transition curve, it is apparent that KL4 experienced a greater upward shift in the index transition temperature( $T_{41J}$ ) after long term heat treatment. It gives the  $T_{41J}$  of -89.5 after ageing in KL4-Ref, compared with -127.8 in normal condition. On the other hand, the index transition temperature was slightly decreased from -1.02 to -3.14 in the KL3.

In order to analyze the fracture behavior, the fracture surfaces of the model alloys were observed by an SEM. Fig. 2 shows the fracture surface of the KL3 and KL4 in the lower transition region. In the SEM observation results, the fracture behavior of the KL4 changed from a transgranular to a partially intergranular manner after

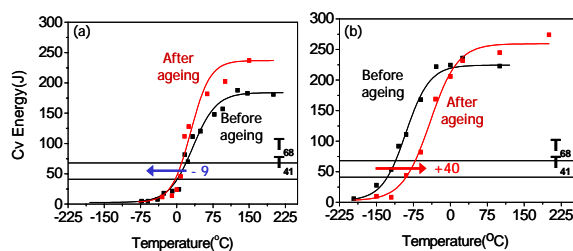


Fig. 1 Charpy transition curves of the  
(a) KL3 and (b) KL4

a long-term heat treatment. However, the fracture appearance of the KL3 did not show any intergranular behavior in either condition. Based on the mechanical test and fracture surface analysis results, The KL4 was embrittled after the long-term heat treatment while KL3 showed little embrittlement behavior in spite of the same P contents.

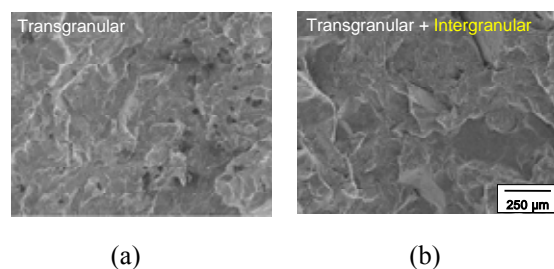


Fig. 2 Fracture analysis of model alloys  
After ageing (a)KL3 and (b)KL4

It is generally known that the cause of temper embrittlement is a grain boundary segregation of the impurity elements such as P and Sb. In addition, the segregation is thermodynamically favored to occur at high angle boundaries, while the boundaries it does not segregate in low energy boundaries such as low angle or coincidence site lattice(CSL) boundaries [5]. In order to compare the boundary features between KL3 and KL4, the misorientation angle distributions of KL3 and KL4 were measured by EBSD. Fig. 3 shows the analysis of the misorientation angles of the KL3 and KL4. In the case of KL3, which has tempered bainitic structure, the fraction of high angle boundaries (higher than  $10^\circ$ ) is 64.4%. From the misorientation map, we observed that the misorientation angle of the prior austenite grain boundaries were mainly distributed about  $20\sim 63^\circ$  (33.5%), thus 30.9% of high angle boundaries were distributed inside the prior austenite grains in KL3. Though the CSL boundaries in these angle ranges were also detected, its amounts were much smaller than those of misorientation angles. In the case of tempered martensitic KL4, the fraction of high angle boundaries (higher than  $10^\circ$ ) are 47.1%, and the portion of the prior austenite grain boundaries ( $20\sim 55^\circ$ ) was 14.4%. Hence, the 32.7% of high angle grain boundaries were distributed inside the prior austenite

grains. This portion seemed similar to KL3. However, there was a large amount of misorientation angles over the misorientation angle of  $55^\circ$  in KL4 (28.4%), which corresponds to the  $\Sigma 3$  boundary. In addition to this, the number of  $\Sigma 3$  accounted for 90% of misorientation angles over  $55^\circ$ , thus the portion of high energy boundaries in KL4 was only 7%.

Based on the EBSD analysis results, there was a larger portion of high energy boundaries inside the prior austenite grains in the tempered bainitic structure (KL3) than those of the tempered martensitic structure (KL4). Thus the segregation behavior of P in the prior austenite grain boundaries would be much more reduced in KL3 than KL4 after ageing, because the large amounts of segregation occurred in high energy boundaries inside the prior austenite grains.

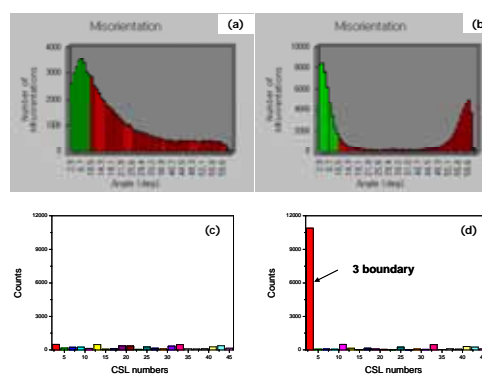


Fig. 3 Histograms of misorientation angles and CSL of (a), (c): KL3 and (b), (d) KL4

#### 4. Summary

In this study, comparison of the temper embrittlement behaviors on SA508 Gr.3 and SA508 Gr.4N low alloy steel by a mechanical test and a microstructural analysis was carried out. The temper embrittlement is occurred in KL4, which has a tempered martensitic structure. The differences in temper embrittlement behavior in two alloys are mainly caused by different portion of high energy boundaries inside the prior austenite grains.

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